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IS 3685 (1966): Methods of chemical analysis of brasses  
[MTD 8: Copper and Copper Alloys]



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*Indian Standard*  
**METHODS OF  
CHEMICAL ANALYSIS OF BRASSES**

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**BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002**

**Gr 7**

*January 1967*

# Indian Standard

## METHODS OF CHEMICAL ANALYSIS OF BRASSES

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# *Indian Standard*

## METHODS OF CHEMICAL ANALYSIS OF BRASSES

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 5 August 1966, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

**0.2** The methods of chemical analysis of brasses, bronzes and other copper base alloys were covered earlier in IS : 441-1955\*. With the revision of specifications on brasses, bronzes and publication of some specifications on copper base alloys, the Sectional Committee felt the need to review these methods and cover them under separate standards to make them more specific. This standard is one of the series and covers the analysis of brasses only.

**0.3** The methods of analysis prescribed in this standard may primarily serve as referee methods and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

**0.4** In the formulation of this standard due weightage has been given to international co-ordination among the standards and the practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

1964 Book of ASTM methods for chemical analysis of metals. Part 32. American Society for Testing and Materials.

FURMAN ( N H ), ED and SCOTT ( W W ). Standard methods of chemical analysis. 1962. Ed 5. D. Van Nostrand Company, Inc., New York.

HILLEBRAND ( W F ), LUNDELL ( G E F ) and BRIGHT ( H A ). Applied inorganic analysis. 1955. Ed 2. John Wiley and Sons, New York.

**0.5** In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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\*Methods for chemical analysis of brasses and bronzes. ( Since withdrawn ).

†Rules for rounding off numerical values ( revised ).



## 1. SCOPE

**1.1** This standard prescribes methods for determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc, and arsenic in the ranges as specified in the relevant Indian Standards on brasses.

## 2. SAMPLING

**2.1** Samples shall be drawn and prepared in accordance with IS : 1817-1961\*.

## 3. QUALITY OF REAGENTS

**3.1** Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1961†) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 4. GENERAL

**4.1 Use of Filter Papers** — In some of the methods prescribed in this standard, relative numbers of Whatman filter papers, which are commonly used, have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

## 5. DETERMINATION OF COPPER AND LEAD SIMULTANEOUSLY BY ELECTROLYTIC METHOD

**5.1 Outline of the Method** — The sample is dissolved in nitric acid and copper and lead are electrolytically deposited and weighed. When the weight of lead peroxide is too small for weighing, lead is estimated colorimetrically as sulphide in presence of sucrose.

### 5.2 Apparatus

**5.2.1 Electrodes** — The following platinum electrodes (see Fig. 1) are recommended, but strict adherence to the shape and size of the electrodes is not essential. For agitation of electrolyte in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed.

**5.2.2 Cathode** — It may be formed either from plain or from perforated sheet or from wire gauze.

\*Methods of sampling non-ferrous metals for chemical analysis.

†Specification for water, distilled quality (revised).

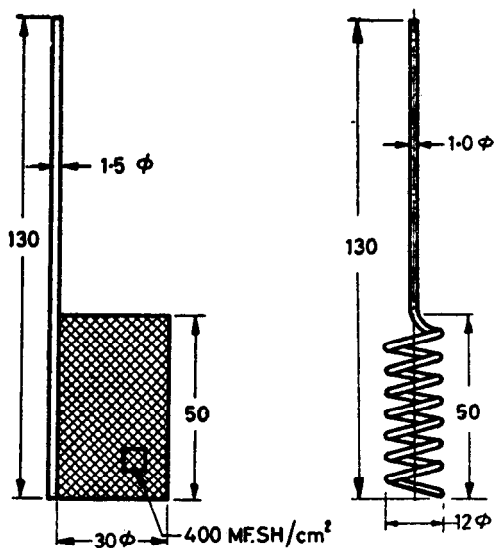


FIG. 1 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

**5.2.2.1** Gauze cathodes preferably made from gauze containing 400 mesh/cm<sup>2</sup> should be used. The wire used for making gauze should be approximately 0.20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about 3 mm on the top and the bottom or by reinforcing the gauze at the top and bottom with platinum ring or band.

**5.2.2.2** The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1.5 mm. It should be flattened and welded throughout the entire length of the gauze. The overall height of the cathode should be approximately 130 mm.

**5.2.3 Anode** — When the amount of lead in the sample is less than 4 mg, a spiral anode should be used. It should be made from 1.0 mm or larger platinum wire formed into spiral of seven turns with a height of approximately 50 mm and diameter of 12 mm, the overall height being 130 mm.

**5.2.3.1** When the amount of lead in the sample is more than 4 mg, the gauze anode should be used. It should be made of the same material and of the same general design as platinum gauze cathode mentioned under 5.2.2. It should be approximately 12 mm in diameter and 50 mm in height, the overall height being 130 mm.

### 5.3 Reagents

**5.3.1 Dilute Nitric Acid** — 1 : 1 and 1 : 99 (v/v).

**5.3.2 Hydrobromic Acid** — 48 percent.

**5.3.3 Dilute Hydrochloric Acid** — 0.1 N approximately.

**5.3.4 Hydrogen Sulphide Solution** — Saturate dilute sulphuric acid (1 : 99) with hydrogen sulphide gas. Prepare fresh as needed.

**5.3.5 Urea** — solid.

**5.3.6 Sulphamic Acid** — solid.

**5.3.7 Ethanol or Methanol** — 95 percent (v/v).

**5.3.8 Lead-Acid Solution** — Dissolve 0.5 g of lead nitrate in 200 ml of water and add with stirring 5 ml of concentrated sulphuric acid to the solution. Allow to stand for 24 hours and siphon or decant through a fine filter paper. Discard the precipitate.

**5.3.9 Hydrogen Peroxide Solution** — 3 percent.

**5.3.10 Ammonium Acetate Solution** — Dissolve 23 g of ammonium acetate in water, and 3 ml of glacial acetic acid and dilute to 100 ml (approximately 3 N).

**5.3.11 Sucrose Solution** — 50 percent (w/v).

**5.3.12 Standard Lead Acetate Solution** — (1 ml = 0.0001 g of lead). Dissolve 0.1831 g of lead acetate [ $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ ] in water containing a little acetic acid, and dilute to one litre.

## 5.4 Procedure

**5.4.1** Dissolve 2 to 5 g of an accurately weighed sample in 25 ml of dilute nitric acid (1 : 1). Heat on a steam bath and boil to expel brown fumes. Add 50 ml of hot water and allow to stand on a steam bath for one hour. If there is any opalescence or precipitate follow the procedure given in 5.4.2.

**5.4.2** Add paper pulp and filter, washing the filter several times with dilute nitric acid (1 : 99). Reserve the filtrate. Transfer the residue to a crucible and ignite completely. Add 10 to 12 ml of hydrobromic acid, evaporate to dryness and ignite. Take up the residue with dilute nitric acid (1 : 1) and boil to expel brown fumes and add to the reserved filtrate.

**5.4.3** Adjust the volume of the solution to 150 ml and add 0.5 g of urea or 0.1 g of sulphamic acid, boil for few minutes. Add one drop of dilute hydrochloric acid. Insert the electrodes, cover the beaker with split watch glasses. Electrolyse with a current of 5 A/dm<sup>2</sup> with constant stirring. When the solution is colourless, wash down the cover glasses, electrodes and sides of the beaker, raising the level of liquid slightly; continue passing the current noting whether or not copper is being plated on the newly exposed surface of platinum cathode. If no copper appears,

transfer about one millilitre of the solution to a spot plate and add a few drops of freshly prepared acidified hydrogen sulphide solution.

**5.4.4** As soon as the deposition is complete, without stopping the current, lower the beaker slowly while washing the cathode with water. Remove the cathode, rinse it with water and then dip in two successive baths of ethanol or methanol.

**5.4.5** Dry for 3 to 5 minutes in an oven at 105° to 110°C, cool and weigh the deposit immediately as metallic copper. Dry the anode in an oven at 110° to 120°C for 30 minutes; the deposit being fragile should be handled with care. Cool the anode and weigh as lead peroxide. If manganese is present, it may co-deposit with lead peroxide. In that case follow the procedure given under 5.4.6.

**5.4.6** Transfer it to a 250-ml beaker, dissolve the deposit in dilute nitric acid (1 : 1) containing a few drops of ethanol, wash the anode thoroughly, and remove it from the beaker. Add 35 ml of 'lead acid' solution and evaporate on the hot plate to dense white fumes. Allow to cool, add 30 ml of water, bring to a boil and allow to stand for 1 to 2 hours. Filter the solution through a tared asbestos Gooch crucible and wash the precipitate with cool lead acid. Heat the precipitate of lead sulphate gently at first, and then to constant weight at very dull redness (500° to 600°C), by suspending the crucible in a larger one so that an air space separates the two.

#### 5.4.7 Calculation

$$\text{Copper, percent} = \frac{A}{D} \times 100$$

$$\text{Lead, percent} = \frac{B \times 0.866}{D} \times 100$$

$$\text{Lead, percent} = \frac{C \times 0.6833}{D} \times 100 \quad (\text{lead determined in accordance with 5.4.6})$$

where

$A$  = weight in g of the copper deposit,

$B$  = weight in g of the lead deposit,

$C$  = weight in g of lead sulphate obtained in 5.4.6, and

$D$  = weight in g of the sample taken.

**5.4.8** When the weight of lead peroxide is small (lead content 0.1 and below) for weighing, proceed as under **5.4.8.1** to determine lead colorimetrically.

**5.4.8.1** Dissolve the lead proxide in 5 ml of dilute nitric acid (1:1) and a few drops of hydrogen peroxide solution. Evaporate to dryness and take up with water. Cool, transfer to a 100-ml graduated flask dilute to the mark and mix well. Pipette a suitable aliquot portion of this solution into a 100-ml Nessler tube, and 6 ml of ammonium acetate solution, 3 drops of dilute nitric acid (1:1), one millilitre sucrose solution and finally 50 ml of water. Add 5 ml of freshly prepared acidified hydrogen sulphide solution, mix well and compare the colour against those of a series of standards. Take a measured volume of standard lead acetate solution to which ammonium acetate solution, dilute nitric acid (1:1), sucrose solution and acidified hydrogen sulphide solution, have been added in same quantities as in the test solution. Finally dilute the standard solution and the test solution to the same volume and compare in a suitable type of colorimeter. It is important to add the acidified hydrogen sulphide solution simultaneously to the standard and test solutions.

#### 5.4.9 Calculation

$$\text{Lead, percent} = \frac{A \times H_1}{H_2} \times \frac{0.01}{B}$$

where

$A$  = volume in ml of the standard lead solution used,

$H_1$  = height of column of standard lead solution,

$H_2$  = height of column of test solution, and

$B$  = weight in g of the sample taken.

## 6. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

**6.1 Outline of the Method** — Tin is collected and separated by precipitation with ammonium hydroxide in presence of iron. After reduction of the solution tin is determined iodimetrically.

### 6.2 Reagents

**6.2.1 Concentrated Hydrochloric Acid** — sp gr 1.16 (conforming to IS: 265-1950\*).

**6.2.2 Dilute Nitric Acid** — 1:1 (v/v).

\*Specification for hydrochloric acid (Since revised).

**6.2.3 Ferric Nitrate Solution** — Dissolve 120 g of ferric nitrate crystals [ $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ] in water and dilute to one litre.

**6.2.4 Concentrated Ammonium Hydroxide** — 20 percent.

**6.2.5 Ammonium Chloride Solution** — Dissolve 20 g of ammonium chloride in 500 ml of hydrochloric acid and dilute to one litre with water.

**6.2.6 Antimony Chloride Solution** — Dissolve 20 g of antimony chloride in water and dilute to one litre.

**6.2.7 Iron** — relatively pure iron (not less than 99.85 percent in the form of wire or strip).

**6.2.8 Potassium Iodide Solution** — Dissolve 100 g of potassium iodide in water and dilute to one litre.

**6.2.9 Starch Solution** — Make a suspension of one gram of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh as needed.

**6.2.10 Standard Potassium Iodate Solution** — (1 g = 0.003 g of Sn, approximately 0.05 N). Twice re-crystallize potassium iodate from water and dry at 180°C to constant weight. Dissolve 1.7835 g of the potassium iodate in 200 ml of water containing 1 g of sodium hydroxide and add 10 g of potassium iodide. When solution is complete, dilute to one litre in a volumetric flask. Standardize the solution against pure tin following the procedure described under 6.3.

**6.2.11 Standard Tin Solution** — Dissolve 0.5 g of tin in a covered 400-ml beaker in 300 ml of dilute hydrochloric acid (1:1) by warming gently until the metal has dissolved. If the solution is difficult, add 0.05 to 0.1 g of potassium chlorate. Cool and dilute to one litre in a volumetric flask.

## 6.3 Procedure

**6.3.1** Transfer 2 g of an accurately weighed sample into a 250-ml beaker (in case tin content is 0.1 percent or below, 10 g of sample should be taken). Add 5 ml of concentrated hydrochloric acid and 20 ml of dilute nitric acid, adding more concentrated hydrochloric acid, if necessary, to keep the tin in solution. When the solution is complete, add 10 ml of ferric nitrate solution and boil for 2 minutes. Dilute to 400 ml with water and add concentrated ammonium hydroxide until the precipitated copper hydroxide is redissolved, and the smell of ammonia persists. Heat to boiling and allow to settle for one hour. Filter through a rapid filter paper and wash with hot ammonium chloride solution. Dissolve the precipitate with hot concentrated hydrochloric acid. Re-precipitate with concentrated ammonium hydroxide, boil, filter and wash thoroughly with hot ammonium chloride solution. Remove the paper and precipitate from the funnel and place them in a 500-ml Erlenmeyer flask.

**6.3.2** Add 200 ml of water to the Erlenmeyer flask containing the paper and the precipitate. Add 75 ml of concentrated hydrochloric acid, 2 drops of antimony chloride solution and 5 g of iron and swirl the flask to break up the paper and to aid in the solution of the precipitate. Stopper the flask with a three holed rubber stopper containing an inlet tube for carbon dioxide, an air condenser, and a hole for the burette (glass plugged) (see Fig. 2). During reduction a very slow stream of carbon dioxide shall be passed through the flask. Heat the solution to boiling, and boil with continuous evolution of gas for at least 45 minutes. After reduction is complete, cool the contents of the flask to about 10°C, maintaining an atmosphere of carbon dioxide by passing carbon dioxide gas into the flask. Add 5 ml of potassium iodide solution and 5 ml of starch solution and titrate with the standard potassium iodate solution to persistent blue colour.

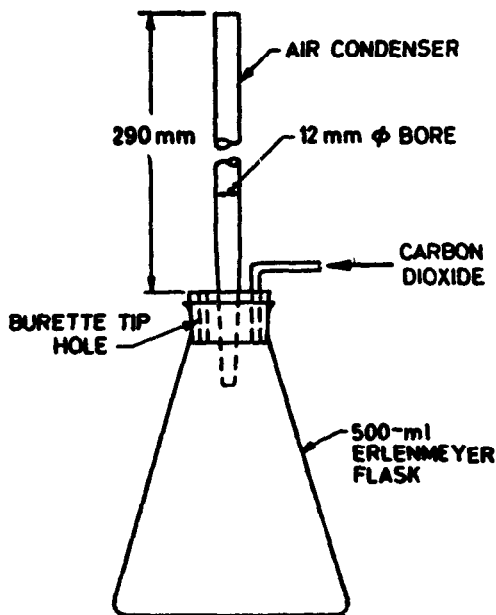


FIG. 2 APPARATUS FOR REDUCTION OF TIN

**6.3.2.1** Carry out a blank determination following the same procedure and using the same quantities of all the reagents but without the sample.

#### 6.4 Calculation

$$\text{Tin, percent} = \frac{(A - B) C}{D} \times 100$$

where

*A* = volume in ml of the standard potassium iodate solution required for the sample,

*B* = volume in ml of standard potassium iodate solution required for the blank,

*C* = tin equivalent of the standard potassium iodate solution in g/ml, and

*D* = weight in g of the sample taken.

## 7. DETERMINATION OF MANGANESE BY THE BISMUTHATE METHOD

**7.1 Outline of the Method** — The method consists of oxidizing bivalent manganese to permanganic acid by the use of sodium bismuthate in nitric acid solution. The permanganate formed is reduced by ferrous ammonium sulphate solution, excess of which is back titrated with standard permanganate solution.

### 7.2 Reagents

**7.2.1 Dilute Nitric Acid** — 3 : 7 and 3 : 97 (*v/v*) free from nitrous acid.

**7.2.2 Sodium Bismuthate** — containing approximately 80 percent of sodium bismuthate ( $\text{NaBiO}_3$ ).

**7.2.3 Ferrous Ammonium Sulphate Solution** — Dissolve 12 g of ferrous ammonium sulphate in one litre of cold dilute sulphuric acid (5 : 95).

**7.2.4 Phosphoric Acid** — 85 percent (*w/v*).

**7.2.5 Standard Sodium Oxalate Solution** — 0.03 N. Dissolve 2.01 g of sodium oxalate in water and make up to one litre in a graduated flask.

**7.2.6 Standard Potassium Permanganate Solution** — 0.03 N approximately. Dissolve 0.96 g of potassium permanganate in one litre of water. Let stand in the dark for 2 weeks and filter through a sintered glass crucible avoiding contact with organic material. Store in a dark coloured, glass-stoppered bottle. Standardize against sodium oxalate (*AR*).

**7.3 Procedure** — Place one gram of accurately weighed sample in a 500-ml Erlenmeyer flask, add 50 ml of dilute nitric acid (3 : 7), and heat gently until the reaction is over. Cool and add 0.5 g of sodium bismuthate. Heat for a few minutes until the purple colour has disappeared. Slowly add ferrous ammonium sulphate solution until the precipitated manganese dioxide dissolves and the solution becomes clear. Boil to expel brown fumes. Cool to 10° to 15°C, add one gram of sodium bismuthate (or an amount equal to at least 26 times the weight of manganese present) and



agitate vigorously for one minute. Add 50 ml of cold dilute nitric acid (3:7) and filter through an asbestos Gooch crucible. Wash with 50 ml of cold dilute nitric acid (3:97). The filtrate shall be perfectly clear. Re-filter if the solution is not clear, maintain the temperature of the filtrate between 10° to 15°C. Add 2 ml of phosphoric acid and measure excess of ferrous ammonium sulphate solution. Titrate immediately with standard potassium permanganate solution to the faint pink colour.

**7.3.1** Carry out a blank determination following the same procedure and using the same amount of the ferrous ammonium sulphate solution and other reagents as were used in the titration, but without the sample.

## 7.4 Calculation

$$\text{Manganese, percent} = \frac{(A - B) C}{D} \times 1.1$$

where

*A* = volume in ml of standard potassium permanganate solution required to titrate the blank,

*B* = volume in ml of standard potassium permanganate solution required for the test solution,

*C* = normality of the standard potassium permanganate solution, and

*D* = weight in g of the sample taken.

## 8. DETERMINATION OF PHOSPHORUS BY THE ALKALIMETRIC METHOD

**8.1 Outline of the Method** — Phosphorus is precipitated as ammonium phosphomolybdate filtered, washed and dissolved in excess of sodium hydroxide. The unreacted alkali is back titrated against standard hydrochloric acid.

### 8.2 Reagents

**8.2.1 Concentrated Nitric Acid** — sp gr 1.42 (conforming to IS:264-1960\*).

**8.2.2 Concentrated Hydrochloric Acid** — See 6.2.1.

**8.2.3 Ferric Chloride Solution** — Dissolve 25 g of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) in 400 ml of water, and filter, if the solution is not clear.

\*Specification for nitric acid (*revised*). (Since revised).

**8.2.4 Ammonium Chloride** — solid. Also, a one percent solution (*w/v*).

**8.2.5 Concentrated Ammonium Hydroxide** — 20 percent.

**8.2.6 Dilute Hydrochloric Acid** — 1 : 1 and 2 : 98 (*v/v*).

**8.2.7 Hydrogen Sulphide** — gas.

**8.2.8 Acidified Hydrogen Sulphide Solution** — Dilute 10 ml of concentrated hydrochloric acid with one litre of water and saturate with hydrogen sulphide.

**8.2.9 Ammonium Nitrate** — solid.

**8.2.10 Ammonium Molybdate Solution** — Add solution *A* to solution *B* with constant stirring, then add 0.1 g. of ammonium phosphate dissolved in 10 ml of water and let stand for at least 24 hours before using. Keep the solution in a cool place and always filter before using.

**8.2.10.1** Prepare solutions *A* and *B* as follows:

- a) *Solution A* — Place in a large beaker 135 g of 85 percent molybdic acid, mix it thoroughly with 320 ml of water, add 187 ml of concentrated ammonium hydroxide (20 percent) and 80 ml of concentrated nitric acid.
- b) *Solution B* — Mix 536 ml of concentrated nitric acid and 1 280 ml of water.

**8.2.11 Dilute Nitric Acid** — 2 : 98 (*v/v*).

**8.2.12 Potassium Nitrate Wash Solution** — Dissolve 10 g of potassium nitrate in water and dilute to one litre.

**8.2.13 Sodium Hydroxide Solution** — 0.1 N approximately.

**8.2.14 Phenolphthalein Indicator Solution** — Dissolve 0.5 g of the phenolphthalein in 100 ml of 60 percent alcohol.

**8.2.15 Standard Hydrochloric Acid** — 0.1 N. Dilute about 11 ml of concentrated hydrochloric acid to one litre and standardize against sodium carbonate.

### 8.3 Procedure

**8.3.1** Transfer about 5 g of the accurately weighed sample into a 400-ml beaker and dissolve in a mixture of 20 ml of concentrated nitric acid and 5 ml of concentrated hydrochloric acid. When dissolution is complete, add 10 ml of ferric chloride solution and 5 g of ammonium chloride, dilute the solution to 200 ml, make ammoniacal, and heat to boiling. Filter through a rapid filter paper and wash the beaker and the precipitate with hot ammonium chloride solution.

**8.3.2** Transfer the paper and the precipitate to the original beaker, add 30 ml of dilute hydrochloric acid (1 : 1) and digest on a steam bath until the precipitate is entirely dissolved. Bring the solution to boil, filter through a rapid filter paper and wash ten times with hot dilute hydrochloric acid (2 : 98). Dilute the filtrate to 300 ml and pass hydrogen sulphide gas for 30 minutes. Allow the precipitated sulphide to coagulate for 2 to 3 hours, and filter, using a close texture paper. Wash the precipitate five times with acidified hydrogen sulphide solution. Reserve the precipitate for arsenic determination.

**8.3.3** Evaporate the filtrate to approximately 20 ml and transfer to a 500-ml conical flask. Add 15 ml of concentrated nitric acid, boil gently for 10 minutes and add 100 ml of water. Cool the solution to 45°C and add 5 g of ammonium nitrate. Add 60 ml of the ammonium molybdate solution in small portions, while swirling the flask. Close the flask with a rubber stopper, shake vigorously for 5 minutes, and allow to stand for one hour. Filter, wash the flask and the precipitate three times with dilute nitric acid and then with potassium nitrate wash solution till 10 ml of the filtrate collected in a test tube does not consume more than one drop of sodium hydroxide solution using a drop of phenolphthalein indicator solution (begin testing the filtrate from third wash).

**8.3.4** Transfer the paper and the precipitate to the flask, stopper the flask, add 30 ml of carbon dioxide free water and shake vigorously until the paper is disintegrated. Wash down the stopper and sides of the flask, add 3 or 4 drops of phenolphthalein indicator solution, while shaking the flask, add sodium hydroxide solution from a burette until the pink colour persists and then add 2 ml in excess. Dilute to about 100 ml with water and titrate with standard hydrochloric acid until the pink colour is completely discharged.

**8.3.5** Carry out a blank determination on the same quantity of sodium hydroxide solution which was added to the test, and titrate with standard hydrochloric acid in presence of few drops of phenolphthalein indicator solution.

## 8.4 Calculation

$$\text{Phosphorus, percent} = \frac{(A - B) \times C \times 0.135}{D}$$

where

*A* = volume in ml of the standard hydrochloric acid required for the blank,

*B* = volume in ml of the standard hydrochloric acid required to titrate the excess sodium hydroxide,

*C* = normality of standard hydrochloric acid, and

*D* = weight in g of the sample taken.

## 9. DETERMINATION OF NICKEL BY THE DIMETHYL-GLYOXIME ( GRAVIMETRIC ) METHOD

**9.1 Outline of the Method** — After removal of tin, copper and lead, nickel is precipitated with dimethylglyoxime solution in alkaline medium, filtered, dried and weighed.

### 9.2 Reagent

**9.2.1 Dilute Nitric Acid** — 1 : 1 ( v/v ).

**9.2.2 Concentrated Sulphuric Acid** — sp gr 1.84 ( conforming to IS : 266-1961\* ).

**9.2.3 Dilute Sulphuric Acid** — 3 : 97 ( v/v ).

**9.2.4 Ammonium Chloride** — solid.

**9.2.5 Tartaric Acid Solution** — Dissolve 250 g of tartaric acid in water and dilute to one litre.

**9.2.6 Concentrated Ammonium Hydroxide** — 20 percent.

**9.2.7 Dimethylglyoxime Solution** — Dissolve 10 g of dimethylglyoxime in one litre of rectified spirit ( conforming to IS : 323-1959† ) and filter before using.

**9.2.8 Concentrated Hydrochloric Acid** — See 6.2.1.

### 9.3 Procedure

**9.3.1** Take 2 g of accurately weighed sample, dissolve in 25 ml of dilute nitric acid, evaporate to dryness. Take up the residue with 10 ml of dilute nitric acid, boil and filter off tin. Remove the copper and lead by the electrolytic method as described under 5.4. ( If silicon is present, add 5 ml of concentrated sulphuric acid and take to fumes. Dilute and filter to remove silica. )

**9.3.2** Dilute the solution to 200 ml. Add 5 g of ammonium chloride. Add 10 ml of tartaric acid solution. Neutralize with concentrated ammonium hydroxide and add one millilitre in excess. The solution should remain clear after this treatment. Heat to 70°C and add 0.4 ml of dimethylglyoxime solution for each one milligram of nickel present and add 5 to 10 ml in excess, pouring directly into the solution and not down the beaker wall. Stir the mixture vigorously and allow to cool to room temperature with occasional stirring. Filter through a sintered glass crucible of medium porosity and wash well with water. Dissolve the precipitate in a small amount of concentrated hydrochloric acid and re-precipitate nickel using

\*Specification for sulphuric acid ( revised ).

†Specification for rectified spirit ( revised ).

1 to 2 ml of dimethylglyoxime solution. Dry the precipitate at 120°C to constant weight and weigh as nickel dimethylglyoxime.

#### 9.4 Calculation

$$\text{Nickel, percent} = \frac{A}{B} \times 20.32$$

where

$A$  = weight in g of nickel dimethylglyoxime, and

$B$  = weight in g of the sample taken.

### 10. DETERMINATION OF IRON

**10.1 Outline of the Method** — Iron is reduced in a boiling solution with stannous chloride the excess of which is destroyed with mercuric chloride. The reduced iron is titrated with standard potassium dichromate solution using sodium diphenylamine sulphonate as internal indicator for lower iron contents, iron is estimated colorimetrically with thiocyanate.

#### 10.2 Reagents

**10.2.1 Dilute Nitric Acid** — 1 : 1 and 1 : 99 ( $v/v$ ).

**10.2.2 Concentrated Nitric Acid** — See 8.2.1.

**10.2.3 Perchloric Acid** — 70 percent ( $v/v$ ).

**10.2.4 Hydrobromic Acid** — 48 percent ( $v/v$ ).

**10.2.5 Concentrated Ammonium Hydroxide** — 20 percent.

**10.2.6 Ammonium Chloride Solution** — 2 percent ( $w/v$ ).

**10.2.7 Dilute Hydrochloric Acid** — 1 : 1 ( $v/v$ ).

**10.2.8 Stannous Chloride Solution** — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

**10.2.9 Mercuric Chloride Solution** — Prepare a saturated solution of mercuric chloride in water.

**10.2.10 Concentrated Sulphuric Acid** — See 9.2.2.

**10.2.11 Sulphuric-Phosphoric Acid Mixture** — Add slowly 150 ml of concentrated sulphuric acid (see 10.2.10) to 700 ml of water with continuous stirring. Add to this 150 ml of syrupy phosphoric acid.

Destroy any oxidizable impurities by adding potassium permanganate solution (0.1 N) drop by drop until the pink colour of permanganate persists, and cool the solution.

**10.2.12 Sodium Diphenylamine Sulphonate Indicator Solution** — Dissolve 0.2 g of sodium diphenylamine sulphonate in 100 ml of hot water

**10.2.13 Standard Potassium Dichromate Solution** — 0.05 N. Dry pure potassium dichromate in an oven at 105° to 110°C for one and a half hours. Cool in a desiccator and transfer exactly 2.4517 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well. Preserve the solution in an amber-coloured bottle.

**10.2.14 Potassium Thiocyanate Solution** — Dissolve 97 g of potassium thiocyanate in 200 ml of water and dilute to one litre.

**10.2.15 Ammonium Alum Solution** — Weigh 0.05 g of pure alumina prepared by igniting ammonium alum  $[\text{Al}_2(\text{NH}_4)_2(\text{SO}_4)_4 \cdot 24\text{H}_2\text{O}]$  in a platinum dish, and fuse it with five grams of potassium bisulphate. Extract the cold cake with water, add 10 ml of concentrated sulphuric acid and make up to one litre. This solution is required as a buffer to counteract the effect of the aluminium sulphate in the test solution on the tint of the ferric thiocyanate.

**10.2.16 Standard Iron Solution** — (1 ml = 0.0001 g of Fe). Dissolve 0.7024 g of ferrous ammonium sulphate  $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$  in water, add five millilitres of concentrated sulphuric acid and oxidize with bromine water. Boil to expel excess bromine, cool and make up to one litre.

### 10.3 Procedure [By the Potassium Dichromate (Volumetric) Method for Iron Content >0.1 Percent]

**10.3.1** Dissolve 5 g of accurately weighed sample free from adventitious iron in 50 ml of dilute nitric acid (1:1) in a 250-ml beaker and heat to expel brown fumes. Dilute the solution to 100 ml and digest on a steam bath for one hour. Filter off the metastannic acid and wash with hot dilute nitric acid (1:99) and reserve the filtrate.

**10.3.2** Transfer the paper to the original beaker. Add 15 ml of concentrated nitric acid and 10 ml of perchloric acid and heat gently until white fumes appear. Cool and wash down the sides of the beaker. Add 10 ml of hydrobromic acid and boil gently until white fumes appear. Repeat hydrobromic acid treatment to remove last traces of tin. Dilute and heat to dissolve the separated salts. Filter off any silica and combine it with the filtrate obtained in **10.3.1**.

**10.3.3** Adjust the volume of the solution to 150 ml and add concentrated ammonium hydroxide until slightly but distinctly alkaline. Boil for a few minutes and allow to settle for some time. Filter while hot, using a medium-texture paper and wash a few times alternately with ammonium chloride solution and hot water. Dissolve the precipitate through the filter paper with 20 ml of hot dilute hydrochloric acid and wash thoroughly with hot water, collecting the solution and washing in the original beaker.

**10.3.4** Evaporate the solution to 15 to 20 ml. Heat to boiling and to the boiling solution add stannous chloride solution drop by drop with continuous stirring until the solution just becomes colourless. Add two to three drops excess of stannous chloride solution and wash down the sides with a little water. Cool the flask rapidly under running tap water until the contents have cooled down to room temperature. Dilute to 250 ml.

**10.3.5** Add excess of mercuric chloride solution (about five millilitres) in one instalment and shake the flask. At this stage a silky white precipitate appears. If it fails to appear or if a blackish precipitate appears, reject the test. If the precipitate is too much, add further five millilitres of mercuric chloride solution. Keep for about two to five minutes and add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 250 ml, washing down the sides of the flask. Add three to four drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until the colour begins to darken. Continue the titration slowly till a drop of the dichromate solution produces a stable violet-blue colouration.

**10.3.6** Make a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

### 10.3.7 Calculation

$$\text{Iron, percent} = \frac{(A-B) C}{D} \times 5.585$$

where

*A* = volume in ml of standard potassium dichromate solution required for the test solution,

*B* = volume in ml of standard potassium dichromate solution required to titrate the blank,

*C* = normality of the standard potassium dichromate solution, and

*D* = weight in g of the sample taken.

**10.4 Procedure (By the Colorimetric Method for Iron Content  $\leq 0.1$  Percent)**

**10.4.1** Transfer the solution obtained under 10.3.3 to 250-ml measuring flask and make up the volume with water. Take a suitable aliquot to a 100-ml Nessler tube ( $N_1$ ) and add 10 ml of potassium thiocyanate solution. In another 100-ml Nessler tube ( $N_2$ ) take 10 ml of potassium thiocyanate solution and 10 ml of the ammonium alum solution. Add standard iron solution from a burette to the Nessler tube ( $N_2$ ), stirring the contents of the tube thoroughly, until the colours match approximately. Dilute the contents of both the tubes to 100 ml with water and stir. Determine the iron content by either comparing the intensities of colours in a suitable type of colorimeter or with the help of an absorptiometer. If a colorimeter is used, the iron content shall be calculated as given under 10.4.2.

**10.4.2 Calculation**

$$\text{Iron, percent} = A \times \frac{250}{B} \times \frac{0.01}{C} \times \frac{H_1}{H_2}$$

where

$A$  = volume in ml of the standard iron solution used,

$B$  = volume in ml of the aliquot taken,

$C$  = weight in g of the sample taken,

$H_1$  = height of column of the standard solution, and

$H_2$  = height of column of the test solution.

**11. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD**

**11.1 Outline of the Method** — Silica obtained after dehydration of the solution in the sulphuric acid medium is determined by hydrofluorization of residue as usual.

**11.2 Reagents**

**11.2.1 Concentrated Hydrochloric Acid** — See 6.2.1.

**11.2.2 Concentrated Nitric Acid** — See 8.2.1.

**11.2.3 Concentrated Sulphuric Acid** — See 9.2.2.

**11.2.4 Dilute Hydrochloric Acid** — 1 : 5 and 1 : 99 ( $v/v$ ).

**11.2.5 Ammonium Chloride** — solid.

**11.2.6 Dilute Sulphuric Acid** — 1 : 1 ( $v/v$ ).

**11.2.7 Hydrofluoric Acid** — 40 percent.



### 11.3 Procedure

**11.3.1** Take 5 g of an accurately weighed sample to a porcelain vessel and dissolve in 10 ml of concentrated hydrochloric acid and 20 ml of concentrated nitric acid. When solution is complete, add 30 ml of concentrated sulphuric acid. Evaporate on a hot plate until dense white fumes are given off. Allow to cool, add carefully 100 ml of dilute hydrochloric acid (1 : 5) and bring to boil. If lead is present in the alloy, add 5 g of ammonium chloride and continue to boil till lead sulphate is in solution. Filter the solution immediately through No. 40 filter paper, completely transfer the residue to the paper and wash thoroughly with dilute hydrochloric acid (1 : 99). Reserve the residue.

**11.3.2** Return the filtrate to the original vessel and carry out the procedure as given under 11.3.1. Transfer both the papers and residues to a platinum crucible. Ignite at a low temperature until the paper has been burnt off and then at 900° to 950°C cool in a desiccator and weigh to constant weight. Add one or two drops of dilute sulphuric acid and sufficient hydrofluoric acid (about 5 ml) to dissolve the residue and evaporate the solution slowly to dense white fumes. Continue to heat the crucible until all the free sulphur trioxide has been expelled, and then ignite at 900° to 950°C for 5 minutes. Cool in a desiccator and weigh. The loss in weight represents silica.

**11.3.3** Make a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

### 11.4 Calculation

$$\text{Silicon, percent} = \frac{A - B}{C} \times 46.72$$

where

$A$  = weight in g of silica,

$B$  = correction for blank in g, and

$C$  = weight in g of the sample taken.

## 12. DETERMINATION OF ALUMINIUM BY THE OXINE METHOD

**12.1 Outline of the Method** — The solution of the sample is electrolysed in mercury cathode cell and electrolyte separated from the amalgam. Aluminium in the electrolyte is then estimated gravimetrically as oxinate.

### 12.2 Apparatus

**12.2.1 A Mercury Cathode Cell** — The modified Melavan type mercury cathode cell with motor-driven stirrer (see Fig. 3), shall be used.

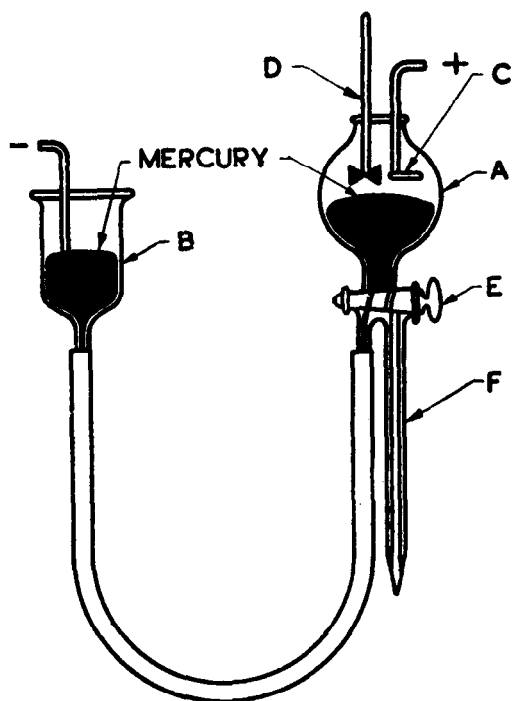


FIG. 3 MELAVAN TYPE MERCURY CATHODE CELL

### 12.3 Reagents

12.3.1 *Concentrated Hydrochloric Acid* — See 6.2.1.

12.3.2 *Concentrated Nitric Acid* — See 8.2.1.

12.3.3 *Dilute Sulphuric Acid* — 1 : 19 (v/v).

12.3.4 *Ferric Chloride Solution* — 10 percent (w/v).

12.3.5 *Concentrated Ammonium Hydroxide* — 20 percent.

12.3.6 *Ammonium Chloride Solution* — 10 percent (w/v).

12.3.7 *Ammonium Acetate Solution* — 40 percent (w/v).

12.3.8 *Bromocresol Purple Indicator Solution* (0.4 g per litre) — Dissolve 0.04 g of bromocresol purple indicator in 100 ml of hot water, cool, and filter.

**12.3.9 Dilute Ammonium Hydroxide — 1 : 3 (v/v).**

**12.3.10 8-Hydroxyquinoline (Oxine) Solution — 2 percent (w/v).** Dissolve five grams of 8-hydroxyquinoline in 15 ml of glacial acetic by warming and dilute with water to 250 ml.

**12.4 Procedure**

**12.4.1** Transfer one to five grams of an accurately weighed sample, to a 250-ml beaker. Add 25 ml of concentrated hydrochloric acid and 5 ml of concentrated nitric acid. Add 50 ml of dilute sulphuric acid. Heat to boiling allow to settle and filter off any lead sulphate. To the filtrate, add 2 ml of ferric chloride solution and concentrated ammonium hydroxide until the solution is distinctly ammoniacal, boil and allow to settle on a steam bath for 5 minutes. Filter on a loose-texture paper, and wash the beaker and precipitate with hot ammonium chloride solution until most of the blue copper salts have been washed out.

**12.4.2** Place the paper and precipitate in the original beaker, add 5 ml of concentrated sulphuric acid and 20 ml of concentrated nitric acid. Heat until all organic matter is destroyed, adding small quantities of concentrated nitric acid as required. Evaporate to dense white fumes. Cool, wash down the cover glass and sides of the beaker with water, and evaporate to dense white fumes again to remove the last traces of concentrated nitric acid. Transfer the solution to the mercury cathode cell, keeping the volume below 100 ml. Ignore the hydrolysis that may take place as shown by the cloudiness of the solution.

**12.4.3** Electrolyse overnight at a current of 8 to 10 amperes. When electrolysis is complete, separate the mercury from the electrolyte while at least a part of the current is kept on. If the electrolyte contains floating particles of loose amalgam after this separation, filter the electrolyte and discard the amalgam.

**12.4.4** To the filtrate add 10 ml of concentrated hydrochloric acid, 15 ml of ammonium acetate solution and 8 to 10 drops of bromocresol purple indicator solution. Add dilute ammonium hydroxide until the indicator becomes distinct purple. While stirring, add slowly from a burette oxine solution using 15 to 20 percent in excess of the amount necessary to precipitate aluminium. Heat the solution and precipitate to boiling while stirring occasionally, maintain the temperature between 60° and 70°C.

**12.4.5** Filter off the precipitate, using a 35-ml fritted-glass crucible of fine porosity, and using moderate suction. Wash the precipitate with 100 ml of cold water, dry for three hours at 130°C, cool in a desiccator and weigh.

## 12.5 Calculation

$$\text{Aluminium, percent} = \frac{A \times 5.87}{B}$$

where

$A$  = weight in g of aluminium oxyquinolate, and

$B$  = weight in g of the sample taken.

## 13. DETERMINATION OF ZINC BY THE EDTA METHOD

**13.1 Outline of the Method** — After the removal of copper and lead, zinc is precipitated as sulphide, filtered, dissolved in sulphuric acid and titrated with EDTA solution.

### 13.2 Reagents

**13.2.1 Dilute Nitric Acid** — 1 : 1 ( $v/v$ ).

**13.2.2 Tartaric Acid Solution** — 30 percent ( $w/v$ ).

**13.2.3 Concentrated Ammonium Hydroxide** — 20 percent.

**13.2.4 Methyl Red Indicator Solution** — 0.1 percent ( $w/v$ ). Dissolve 0.1 g of methyl red in 3.72 ml of sodium hydroxide solution (0.1 N) and dilute to 250 ml with water. Filter, if necessary.

**13.2.5 Formic Acid Mixture** — To 20 ml of formic acid (sp gr 1.20), add 25 g of ammonium sulphate and 3 ml of concentrated ammonium hydroxide and dilute to 100 ml.

**13.2.6 Hydrogen Sulphide** — gas.

**13.2.7 Dilute Sulphuric Acid** — 1 : 1 ( $w/v$ ).

**13.2.8 Sodium Hydroxide Solution** — 200 g/l ( $w/v$ ).

**13.2.9 Buffer Solution** — Dissolve 54 g of ammonium chloride in 300 ml of water, add 350 ml of ammonium hydroxide and dilute to one litre. This solution has a pH of 10.

**13.2.10 Eriochrome Black-T Indicator Solution** — Dissolve 0.4 g of the sodium salt in 20 ml of ethanol, add 30 ml of tri-ethanolamine and store in a polythene-dropping bottle.

**13.2.11 Standard Disodium Ethylenediamine Tetra-acetate (EDTA), Standard Solution (0.05 M)** — Dissolve 18.6 g of the salt in 600 ml of hot water. Cool to room temperature, add 0.1 g of magnesium chloride ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) and dilute to one litre with water. Standardize the solution as follows:

Transfer to an 800-ml beaker an aliquot of the standard zinc solution (see 13.2.12) approximately equal in zinc content to the aliquot of the

sample. Continue as directed in 13.3. Calculate the equivalent of the EDTA solution in terms of grams of zinc per millilitre of solution.

**13.2.12 Standard Zinc Solution** — Dissolve 4.0000 g of pure electrolytic zinc in 200 ml of dilute hydrochloric acid (1 : 4), cool and dilute to one litre in a volumetric flask.

### 13.3 Procedure

**13.3.1** Dissolve one gram of the accurately weighed sample in dilute nitric acid and remove copper, lead and tin (if present) as described under 5.4.2 to 5.4.4.

**13.3.2** Add 25 ml of tartaric acid solution and neutralize with concentrated ammonium hydroxide using methyl red as indicator. Add 25 ml of formic acid mixture, heat to 70° to 80°C. Add a little paper pulp and pass hydrogen sulphide gas rapidly through the solution for 30 minutes. Allow the precipitate of zinc sulphide to coagulate, filter on a pulp pad and wash with warm formic acid wash solution.

**13.3.3** Dissolve the precipitate of zinc sulphide in hot dilute sulphuric acid and collect the solution in a 250-ml volumetric flask. Dilute to the mark.

**13.3.4** Take a suitable aliquot. Neutralize the solution with sodium hydroxide solution using methyl red indicator. Add 30 ml of buffer solution, 5 drops of eriochrome black-T indicator. Titrate slowly with EDTA solution to bluish green end point.

### 13.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

*A* = volume in ml of EDTA solution required for titration of the solution,

*B* = zinc equivalent in grams per millilitre of EDTA solution, and

*C* = weight in g of sample represented by the aliquot.

## 14. DETERMINATION OF ARSENIC BY THE GUT ZEIT METHOD

**14.1 Outline of the Method** — The stains produced on the mercuric bromide paper by arsine evolved by the action of zinc on sulphuric acid solution of the sample are compared with the standard arsenic stains.

**14.2 Reagents** — In addition to the reagents given in IS : 2088-1962\*, the following reagents are required.

**14.2.1 Sodium Hydroxide** — 1 N approximately.

**14.2.2 Dilute Sulphuric Acid** — 5 N approximately.

**14.3 Procedure** — Dissolve the precipitate preserved under 8.3.2 in 20 ml of sodium hydroxide. Neutralize the solution with dilute sulphuric acid. Dilute to 100 ml with water containing 10 ml of dilute sulphuric acid. Take a suitable aliquot of the solution so as to contain 0.01 to 0.05 mg of arsenic and follow the procedure as given in IS : 2088-1962\*.

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\*Modified Gutzeit method of test for arsenic. ( Since revised ).

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